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(FILE 'HOME' ENTERED AT 11:02:26 ON 15 SEP 2003)
FILE 'REGISTRY' ENTERED AT 11:02:41 ON 15 SEP 2003

L1 1 S TEFLON/CN
FILE 'CA' ENTERED AT 11:04:06 ON 15 SEP 2003
L2 53895 S EXTRACT?(5A) (VESSEL OR CONTAINER OR CELL OR TUBE)
L3 105 S L2 AND(L1 OR TEFLON OR PTFE OR POLYTETRAFLUOROETHYLENE OR(POLY OR
POLYMER) (1A)TETRAFLUOROETHYLENE)
L4 1293 S L2 AND FLOW?
L5 25 S L3 AND L4
L6 14016 S (FILTRAT? OR FILTER?) (5A) (VESSEL OR CONTAINER OR CELL OR TUBE)
L7 179 S L6 AND(L1 OR TEFLON OR PTFE OR POLYTETRAFLUOROETHYLENE OR(POLY OR
POLYMER) (1A)TETRAFLUOROETHYLENE)
L8 32 S L7 AND PRESSUR?
L9 57 S L5,L8
L10 53 S (L9 NOT PY>2000)OR(L9 AND PATENT/DT)

=> d l10 bib,ab 1-53

L10 ANSWER 9 OF 53 CA COPYRIGHT 2003 ACS on STN
AN 134:133646 CA
TI Lined sampling **vessel** including a **filter** to separate solids from liquids on
exit
IN Shurtliff, Rodney M.; Klingler, Kerry M.; Turner, Terry D.
PA Bechtel Bwxt Idaho, Llc, USA
SO U.S., 12 pp.
PI US 6187209 B1 20010213 US 2000-533462 20000323
PRAI US 2000-533462 20000323
AB A filtering app. has an open canister with an inlet port. A canister lid is
provided which includes an outlet port for the passage of fluids from the
canister. Liners are also provided which are shaped to fit the interiors of
the canister and the lid, with at least the canister liner preferably being
flexible. The sample to be filtered is positioned inside the canister
liner, with the lid and lid liner being put in place thereafter. A filter
element is located between the sample and the outlet port. Seals are formed
between the canister liner and lid liner, and around the outlet port to
prevent fluid leakage. A **pressure** differential is created between the
canister and the canister liner so that the fluid in the sample is ejected
from the outlet port and the canister liner collapses around the retained
solids.

L10 ANSWER 13 OF 53 CA COPYRIGHT 2003 ACS on STN
AN 132:121580 CA
TI **Flow** injection Fourier transform infrared determination of caffeine in
coffee
AU Bouhsain, Z.; Garrigues, J. M.; Garrigues, S.; de la Guardia, M.
CS Department of Analytical Chemistry, University of Valencia, Valencia, Spain
SO Vibrational Spectroscopy (1999), 21(1-2), 143-150
AB A fully automated procedure was developed for the FTIR spectroscopic detn.
of caffeine in coffee samples. The method involves the online extn. of
caffeine with CHCl3. Samples, weighed inside empty **PTFE** cartridges of 0.5
cm internal diam. (internal diam.) and 1.5 mL vol., were humidified with
four drops of 0.25M NH3. The cartridge was installed in a **flow** manifold, in
which samples were extd. in a closed-**flow** system with 1 mL CHCl3 during 6
min. Four hundred microliters of the **ext.** were introduced in a microflow
cell and absorbance measured as a function of time at 1659 cm-1, with a
baseline established between 1900 and 830 cm-1, thus providing a diagram.

Peak height values of the FI recordings, obtained for samples, were interpolated in an external calibration line established from std. solns. of caffeine in CHCl₃. The method provided a limit of detection (LOD) of 9 mg L⁻¹ caffeine, a relative std. deviation of 0.6% for five independent measurements of a soln. contg. 1 mg ml⁻¹ and a sampling frequency of the whole procedure of 6 h⁻¹. Results obtained for market samples agree well with those found by the official chromatog.-spectrometric method, but involving a drastic redn. of solvents, from the 200 mL ether and 50 mL CHCl₃ required for each sample prepn. by the ref. procedure to <30 mL CHCl₃ necessities for the whole detn. of caffeine in a sample, also including stds. and carrier soln.

L10 ANSWER 16 OF 53 CA COPYRIGHT 2003 ACS on STN

AN 131:96498 CA

TI Method performance of the closed vessel microwave-assisted acid extraction using 50% HNO₃:HCl (3:2) with positive **pressure Teflon** membrane filtration

AU Robbat, Albert, Jr.; Simpson, Robert L., III

CS Department Chemistry, Tufts University, Medford, MA, 02155, USA

SO Fresenius' Journal of Analytical Chemistry (1999), 364(4), 305-312

AB The recovery from soil of 22 metals on the U.S. Environmental Protection Agency's (EPA) Target inorg. analyte list is described. The extn. method was developed to provide a safe, rapid, and anal. reliable means of leaching metals from soils and sediments in one procedure. The influence of digestion matrixes, filtration media, ref. std. types, and instrument performance of ICP-AES is presented. The method employs a closed vessel, temp. and **pressure** controlled, microwave acid digestion using 20 mL of 50% HNO₃:HCl in a ratio of (3:2). The digestate was filtered through a pos. **pressure Teflon** membrane cartridge. This procedure recovered all metals at concns. equal to or greater than what is possible by EPA standardized methods or other methods published in the literature. Excellent method precision and accuracy was obtained for all metals, esp. Ag and Sb. The pos. **Teflon** membrane filtration system yielded higher and statistically different concns. of Mn, Zn, Cu, Fe, As, Cd, Pb, Ag, and Sb than paper filtration in half the time. These findings were produced from std. ref. soils and soil collected from a hazardous waste site landfill.

L10 ANSWER 23 OF 53 CA COPYRIGHT 2003 ACS on STN

AN 120:326304 CA

TI Automated supercritical fluid extraction system

IN Houck, Raymond K.; Koebler, Douglas J.; Williams, Glen P.; Kato, Kenneth J.; Parks, Robert D.; Bauer, Paul A., Jr.

PA Suprex Corp., USA

SO PCT Int. Appl., 110 pp.

PI WO 9408683 A1 19940428 WO 1993-US9680 19931012

US 5866004 A 19990202 US 1995-524916 19950908

PRAI US 1992-962463 A 19921016

AB A supercrit. fluid extn. system for extg. analytes from samples comprises a holding means, and a moving means which selectively moves **extn. vessels** into an **extn.** chamber; the system also includes a computer for controlling the extn., and a collecting means having a restrictor for controlling the **flow** of analyte. The system can be used to analyze polyarom. hydrocarbons in soil.

L10 ANSWER 25 OF 53 CA COPYRIGHT 2003 ACS on STN

AN 120:137791 CA

TI Apparatus for supercritical fluid extraction

IN Ichinomya, Makoto; Kato, Osamu; Nishida, Hideo; Yutaka, Hideki; Takenaka, Mamoru

PA Kobe Steel Ltd, Japan
SO Jpn. Kokai Tokkyo Koho, 10 pp.
PI JP 05305202 A2 19931119 JP 1992-111600 19920430
PRAI JP 1992-111600 19920430
AB In a supercrit. fluid **extn.** app. comprising an external **vessel** and an inserted internal vessel for contg. substance to be treated by supercrit. fluid (e.g., CO₂) **flowing** in vertical direction, sealing member(s) (e.g., **PTFE**) is arranged between the outer- and inner circumferential surfaces of the internal- and external vessel adjacent to their upper (or lower) end, and guide member(s) softer than the 2 vessels is arranged between the outer- and inner circumferential surfaces of the internal- and external vessel adjacent to their lower (or upper) end for preventing contacting of the 2 vessels. The sealing member(s) has tensile strength ≥ 250 kg/cm², and gas permeability $\geq 10000 \times 10^{-10}$ cm³-mm/(cm²-s-cmHg). The app. is used for extn. of desired substances from solid substances, e.g., natural products, etc.

L10 ANSWER 42 OF 53 CA COPYRIGHT 2003 ACS on STN
AN 102:181779 CA
TI Determination of sulfur in biological samples by vacuum-ultraviolet inductively-coupled plasma atomic emission spectrometry
AU Morita, Masatoshi; Uehiro, Takashi; Fuwa, Keiichiro
CS Natl. Inst. Environ. Stud., Tsukuba, 305, Japan
SO Analytica Chimica Acta (1984), 166, 283-8
AB Std. ref. materials (e.g., serum, hair, liver, mussel, Chlorella, and orchard leaves) were decompd. by high-**pressure** bomb digestion in a **Teflon container** with HNO₃, dild. with water, **filtered**, and the absorbance was measured at 180.7 nm by the title method for S detn.. The detection limit was ~ 10 μ g S/L. The results were satisfactory.

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STN INTERNATIONAL LOGOFF AT 11:14:49 ON 15 SEP 2003=> d his

(FILE 'HOME' ENTERED AT 16:41:12 ON 11 SEP 2003)

FILE 'CA' ENTERED AT 16:41:57 ON 11 SEP 2003

E MANGANINI S/AU

L1 17 S E3-7

E DOHERTY K/AU

L2 6 S E8,E17

L3 22 S L1-2

L4 1 S L1 AND L2

L5 6496 S MICROWAVE(6A) (PROCESSING OR REACTION OR VESSEL OR CONTAINER OR DIGEST?)

L6 1369 S L5 AND(SAMPLE OR SOIL OR SOLID) (6A) (PREPAR? OR DIGEST? OR REACTI? OR FRACTIONA? OR EXTRACT?)

L7 45 S L6 AND(FILTER OR MEMBRANE OR POROUS)

L8 146 S L6 AND(TEFLON OR PTFE OR PFA OR ULTEM OR POLYTETRAFLUOROETHYLENE)

L9 182 S L4,L7-8

L10 146 S L9 NOT PY>2000

L11 2 S L9 NOT L10 AND PATENT/DT

L12 148 S L10-11

=> d bib,ab 1-148 112

L12 ANSWER 1 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 138:162651 CA

TI Sequential processing reaction vessel for chemical fractionation and analysis

IN Manganini, Steven J.; Doherty, Kenneth W.; Hammer, Terence R.; Lancaster,

Bruce A.

PA USA

SO U.S. Pat. Appl. Publ., 13 pp.

PI US 2003031600 A1 20030213 US 2001-771354 20010125

PRAI US 2000-177837P P 20000125

AB A sequential processing reactor vessel and method is disclosed for accelerated extn. and **fractionation** of chem. analytes from complex **solid sample** materials. The device and method provide for sequential **extn.** of elemental constituents from **solid** materials by sequentially contacting target **samples** within a single **reaction** vessel using different reagents at temps. $\leq 150^\circ$. and pressures up to 150 psi to accelerate reactions. The aggressive chem. treatments provided by the disclosed device and method provide for complete **digestion** of **solid samples** in liq. analyte **samples** that can be directly analyzed by conventional spectrometry or other suitable methods. The disclosed device and method provide for efficient **sample** processing and accelerated **reactions** to significantly reduce processing times and costs for elemental anal. of solids while improving accuracy, precision and reliability of results compared to conventional devices and methods. The disclosed device and method are compatible with both conventional convection and radiant heating sources as well as microwave heating and can be readily adapted to marine, geol., environmental, industrial and research solids anal. applications.

L12 ANSWER 14 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 132:269115 CA

TI **Sample preparation** and determination of the element content from low-weight feather samples

AU Vallner, Judit; Posta, J.; Szep, T.; Braun, M.; Balogh, A.; Kiss, F.

CS Department of Environmental Science, Gyorgy Bessenyei College, Nyiregyhaza, 4400, Hung.

SO Toxicological and Environmental Chemistry (1999), 70(3-4), 297-304

AB In examg. heavy metals in bird feathers, birds must be protected by using as little material as possible. However, small **sample** size has generated difficulties in **sample prepn.** Ultrasonic washing was shown to be efficient in eliminating all water-sol. contamination from the surface of feather samples. Ways to avoid previous contamination from the walls of a **Teflon** bomb during **microwave digestion** are discussed. There is no loss of sample, and the memory effect of the bombs is eliminated. Elemental content (Ca, Fe, K, Mg, Mn, Na, Sr) of a homogenized feather powder sample of the wt. of 1 swallow feather was detd. using inductively coupled plasma at. emission spectrometry with impulse sample introduction.

L12 ANSWER 15 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 132:267822 CA

TI Rapid determination of Os isotopic composition by sparging OsO₄ into a magnetic-sector ICP-MS

AU Hassler, D. R.; Peucker-Ehrenbrink, B.; Ravizza, G. E.

CS MIT/Woods Hole Oceanographic Institution (WHOI) Joint Program in Oceanography, Woods Hole, MA, USA

SO Chemical Geology (2000), 166(1-2), 1-14

AB A method is presented for the rapid detn. of Os isotopic compns. and platinum group element (PGE) concns. using ICP-MS, in the same sediment sample split. The method makes use of transfer of volatile OsO₄ by an Ar gas stream into the torch of a magnetic sector ICP-MS for anal. A variety of partial or complete sample dissoln. methods can be used, namely (1) **microwave oven digestion** of **samples** in pressurized **Teflon** vessels under oxidizing acidic conditions, (2) NiS fire assay for complete sample dissoln. and PGE preconcn., (3) acid leaching techniques to ext. labile PGE

fractions, and (4) Carius tube digestion. **Microwave oven sample digestion** with HF-HNO₃-HCl in an all-**Teflon** system as well as Carius tube digestion also allows the detn. of Os isotopic compn. and Os and Re concns. on the same sample split. Spike-sample equilibration can be monitored online during sparging into the ICP-MS. The elimination of a nebulizer for Os isotope ratio measurements minimizes memory problems that are often assocd. with the detn. of Os isotope ratios in liq. samples by ICP-MS. The external reproducibility of 187Os/188Os measurements of an inhouse Os std. soln. using a single-collector, magnetic sector ICP-MS (Finnigan ELEMENT) is 0.78% (1 S.D., n=13) for analyte amts. ranging from 81 pg to 1.22 ng total Os. The ion yield for 7-min data acquisitions is $\sim 5 \times 10^{-5}$. Compared to neg. thermal ionization mass spectrometry (N-TIMS), sample throughput is about on an order of magnitude higher. If NiS fire assay is used for PGE preconcn., complementary PGE can be detd. in the liq. residue by ICP-MS using a microconcentric nebulizer after the extn. of OsO₄.

L12 ANSWER 17 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 132:147685 CA

TI Usefulness of enzymatic hydrolysis procedures based on the use of pronase E as sample pre-treatment for multi-element determination in biological materials

AU Bermejo-Barrera, Pilar; Fernandez-Nocelo, Susana; Moreda-Pineiro, Antonio; Bermejo-Barrera, Adela

CS Faculty of Chemistry, Nutrition and Bromatology, Department of Analytical Chemistry, University of Santiago de Compostela, Santiago de Compostela, 15706, Spain

SO Journal of Analytical Atomic Spectrometry (1999), 14(12), 1893-1900

AB Several minor (Cu, Fe, Mg and Zn) and trace (Ag, As, Cd and Pb) elements were extd. from biol. materials, such as human hair and mussel, using an enzymic hydrolysis procedure based on pronase E. The reaction conditions, viz., pH and temp., were fixed at optimum values of 7.4 and 37°, resp. Other variables affecting the enzymic hydrolysis procedure, such as enzymic hydrolysis time, enzyme concn., vol. of enzyme soln. and sample mass, were studied and optimized. The pH value was adjusted with a TRIS-HCl soln. as buffer system. The minor elements were measured by FAAS while trace elements were detd. by ETAAS under optimum conditions. In order to det. the total element concn. in **samples**, a **microwave-induced acid digestion** procedure in lab.-made low pressure **PTFE** bombs was optimized. The enzymic hydrolysis was effective for mussel samples (recoveries of about 100% were obtained for As, Cd, Cu and Mg); however, it was poor for human hair (recoveries were lower than 70%).

L12 ANSWER 19 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 131:348663 CA

TI **Sample preparation** by focused **microwave digestion** for ICP-MS

AU Curdova, Eva; Koplik, Richard

CS VSCHT, Prague, 16628, Czech Rep.

SO CHEMagazin (1999), 9(5), 15-16

LA Czech

AB **Microwave-assisted pressure digestion** is a well established method of **sample prepn.** for trace element anal. A new generation of focused microwave decompn. units is represented by the Polish UniClever system. Sample decompn. takes place in a **teflon** (TFM-**PTFE**) vessel under controlled pressure. Temp. and pressure valves are monitored in each individual unit. The whole system can be controlled by a personal computer. **Microwave digestion** in the UniClever system using HNO₂+H₂O₂ mixt. was used for the decompn. of several certified ref. materials of plant origin (alfalfa, rye grass, pine needles, apple leaves). The final detn. of trace elements (Cd,

Cu, Pb, Zn) was accomplished by ICP-MS. The results proved a good efficiency of the sample decompn. and excellent accuracy.

L12 ANSWER 20 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 131:256449 CA

TI **Microwave-Enhanced Flow System for High-Temperature Digestion of Resistant Organic Materials**

AU Pichler, Ulrike; Haase, Anja; Knapp, Guenter; Michaelis, Markus

CS Institute of Analytical Chemistry Micro- and Radiochemistry, Technical University Graz, Graz, A-8010, Austria

SO Analytical Chemistry (1999), 71(18), 4050-4055

AB **Microwave-assisted flow digestion** systems open up new possibilities in fully automated **sample prepn.** for element anal. For an extensive and fast oxidn. of org. materials with nitric acid, temps. of more than 200°C are necessary. To achieve the desired temps. of ~250°C, it is essential that the pressure in the system can be increased up to 35 bar. The **teflon** tubes used, however, do not withstand the vapor pressure of the digestion mixt. at these temp. levels. A high-pressure flow digestion device is described that enables the application of such high temps. by means of a novel pressure equilibration system. **PTFE** or **PFA** tubes can be used up to 250°C if no mech. stress is applied to the tube wall. The pressure equilibration system keeps the pressure inside and outside the digestion tube equal even for extremely fast oxidn. reactions. The digestion of easy, medium, and difficult oxidizable substances (glucose, glycine, and phenylalanine, resp.) shows the importance of digestion temps. around 250°C. The extremely violent digestion of glucose with concd. nitric acid can be carried out as easy as the difficult oxidn. of phenylalanine by means of this system. The SRM TORT 2 (defatted lobster hepatopancreas tissue) was digested under different conditions to indicate the high oxidn. capabilities in comparison with a com. medium-pressure flow digestion device. The anal. of the SRMs milk powder (BCR 063, BCR 151), bovine liver (BCR185), and pig kidney (BCR 186) after digestion at 245°C and 5 min showed good agreement with the certified values.

L12 ANSWER 25 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 131:115534 CA

TI Presence and distribution of arsenical species in beers

AU Herce-Pagliai, C.; Gonzalez, G.; Camean, A. M.; Repetto, M.

CS Departamento de Bioquímica, Bromatología y Toxicología, Universidad de Sevilla, Sevilla, 41012, Spain

SO Food Additives and Contaminants (1999), 16(6), 267-271

AB The total content of arsenic and of its inorg. (As(III) and As(V)) and org. (monomethylarsonic acid, MMAA, and dimethylarsinic acid, DMAA) species were detd. in a set of 21 alc. and alc.-free beer samples using the technique of Hydride Generation At. Absorption Spectrometry. For total arsenic anal., beer **samples** were dried and then **microwave digested** with nitric acid in **polytetrafluoroethylene containers**. For the speciation anal., beers were previously subjected to ion exchange chromatog. to elute the mentioned inorg. and org. arsenical species. Both **microwave digestion** and chromatog. sepn. methods were validated from certified ref. materials and prepd. std. solns., resp. The results obtained are presented in terms of the distribution and occurrence of arsenical species in the samples. The As levels of the beer samples were in the range of 1.5-12.4 µg/L. The influence of the prodn. process for the alc.-free beers in the speciation of arsenic is discussed. In alc. beers MMAA was the most abundant species, and for non-alc. beers inorg. As(III) was similar to the org. species. An estd. intake of total As of 0.47 µg/person/day and 11.4 µg/person/day was obtained for av. consumers and for heavy drinkers, resp.

L12 ANSWER 26 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 131:96498 CA
TI Method performance of the closed **vessel microwave**-assisted acid extraction using 50% HNO₃:HCl (3:2) with positive pressure **Teflon membrane** filtration
AU Robbat, Albert, Jr.; Simpson, Robert L., III
CS Department Chemistry, Tufts University, Medford, MA, 02155, USA
SO Fresenius' Journal of Analytical Chemistry (1999), 364(4), 305-312
AB The recovery from soil of 22 metals on the U.S. Environmental Protection Agency's (EPA) Target inorg. analyte list is described. The extn. method was developed to provide a safe, rapid, and anal. reliable means of leaching metals from soils and sediments in one procedure. The influence of digestion matrixes, filtration media, ref. std. types, and instrument performance of ICP-AES is presented. The method employs a closed **vessel**, temp. and pressure controlled, **microwave acid digestion** using 20 mL of 50% HNO₃:HCl in a ratio of (3:2). The digestate was filtered through a pos. pressure **Teflon membrane** cartridge. This procedure recovered all metals at concns. equal to or greater than what is possible by EPA standardized methods or other methods published in the literature. Excellent method precision and accuracy was obtained for all metals, esp. Ag and Sb. The pos. **Teflon membrane** filtration system yielded higher and statistically different concns. of Mn, Zn, Cu, Fe, As, Cd, Pb, Ag, and Sb than paper filtration in half the time. These findings were produced from std. ref. soils and soil collected from a hazardous waste site landfill.

L12 ANSWER 27 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 131:22488 CA
TI Determination of chromium in airborne particulate matter by high resolution and laser ablation inductively coupled plasma mass spectrometry
AU Wang, Chu-Fang; Chin, Ching-Jer; Luo, Shen-Kay; Men, Lee-Chung
CS Department of Nuclear Science, National Tsing Hua University, Hsinchu, Taiwan
SO Analytica Chimica Acta (1999), 389(1-3), 257-266
AB An optimized **sample digestion** procedure to det. Cr in airborne particulate matter by inductively coupled plasma mass spectrometry (ICP-MS) is discussed. High resolu. (HR) ICP-MS is used to examine how the added acid mixt. and subsequent evapn. process affect spectroscopic interference. Exptl. results indicated a 2-step high pressure bomb acid digestion (first step with HNO₃ + HClO₄; second step with HF) procedure without evapn. is the optimum pretreatment procedure to det. Cr by ICP-MS. In addn., laser ablation (LA) ICP-MS is used directly to analyze for Cr in airborne particulate matter. Results obtained by LA-ICP-MS correlated well with certified values and values obtained from the conventional acid digestion/HR-ICP-MS method. The Cr detection limit in air particles by HR-ICP-MS with the proposed digestion method is <10 ng/g. Furthermore, LA-ICP-MS provides a rapid, direct anal. technique with a detection limit of 0.05 µg/filter.

L12 ANSWER 31 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 130:109334 CA
TI Optimized **microwave digestion** procedure for cadmium analysis of mussel **samples**
AU Yebra, M. C.; Enriquez, M. F.
CS Department of Analytical Chemistry, Nutrition and Bromatology, Chemistry Faculty, Univ. of Santiago, Santiago de Compostela, 15706, Spain
SO Analisis (1998), 26(7), 261-263
AB A simple and rapid acid **sample digestion** method by **microwave** heating in high-pressure **teflon** bombs is reported for the detn. of cadmium in mussels by flame at. absorption spectrometry (FAAS) coupled online with a flow-

injection preconcn. system. Tests concerning the **digestion** time, the power of the **microwaves** and the amt. of nitric acid were studied with the National Research Council of Canada lobster hepatopancreas marine (Tort-1) as certified ref. material. Using 2 min of digestion time and 2 mL of nitric acid, the complete dissoln. of 0.2 g of freeze-dried sample is possible. The anal. results, obtained with calibration graph as well as by the std. addn. method, were agreed well with the certified value of the ref. material. This procedure was applied to the detn. of cadmium in mussel samples from estuaries in Galicia, Spain.

L12 ANSWER 34 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 130:42756 CA

TI Chemical characterization of municipal solid waste incineration residue: dissolution of elements with a **microwave**-dilute acids **digestion** technique compared to conventional methods

AU Dugenest, S.; Olle, M.; Ribes, A.; Grenier-Loustalot, M. F.

CS Service Central d'Analyse, Centre National de la Recherche Scientifique, Vernaison, 69390, Fr.

SO Analisis (1998), 26(7), 256-260

AB The aim of this work was to evaluate the focused microwave technique in an open system for the rapid dissoln. of elements in fly ash and **filter** cakes, in comparison to conventional wet and dry methods. The method involved 15 min of heating in microwave system in 2 N nitric or hydrochloric acid. It was tested on different types of samples and led to the dissoln. of Ca, Na, K, Zn, Pb, As with 2 N HNO₃ or HCl with Cd. Hg with 2 N HNO₃ for subsequent detn. by at. emission or absorption spectrometry. Thereby this method represents a rapid **sample prepn.** technique for chem. analyses.

L12 ANSWER 35 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 129:269679 CA

TI Combined **vessel** system used with **microwave** decomposing apparatus

IN Kubovics, Ferenc

PA Hung.

SO PCT Int. Appl., 16 pp.

PI WO 9845680 A1 19981015 WO 1998-HU30 19980324

PRAI HU 1997-736 19970404

AB The invention relates to a combined **vessel** system used with **microwave** decompn. app. comprising a basic **vessel** placed on a rotary plate for receiving the material to be decompd. - the sample and chem. agents used for decompn. In the combined vessel system the basic vessel is formed to be suitable for a combined microwave decompn. The basic vessel is provided with a bell-shaped refrigerating cover closing the basic vessel from above and having an aperture on its end opposite to the basic vessel. Said aperture of the bell-shaped refrigerating cover is connected with a vacuum pipe draining the gases developed during the decompn.

L12 ANSWER 40 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 128:42984 CA

TI Sample decomposition-Quo vadis

AU Knapp, Gunter

CS Department Analytical Chemistry Micro- and Radiochemistry, Technical University Graz, Graz, Austria

SO Contemporary State and Trends of Decomposition Methods in Analytical Chemistry, Proceedings, Slovak-Austrian Symposium, Kosice, Slovakia, Feb., 1997 (1997), 10-17. Editor(s): Krakovska, Erika. Publisher: Stroffek Publishing, Kosice, Slovakia.

AB A review, with 12 refs., is given on **sample prepn.** techniques particularly decompn. **Sample** decompn. is the most significant **sample prepn.** technique

for trace element anal. Important functions of **sample digestion** are dissoln. of **solid** materials and oxidn. of org. matrix constituents. The detn. of elements in the trace and ultratrace range make great demands on decompn. methods. With respect to correct anal. results the classical decompn. techniques fusion, oxidn. with oxygen and wet digestion were improved to reduce systematic errors caused by contamination or losses of elements. In anal. chem. quality control becomes more significant increasingly. But presently it is applied however, 1st of all to measuring techniques and not to **sample prepn.** One of the most important **sample prepn.** methods in element anal. is **microwave** assisted wet **digestion** in closed pressurized **vessels**. The parameters important for quality control are in this case pressure and temp. Pressure control is important because the closed vessels are tight only up to a certain pressure. During the decompn. procedure this pressure may be exceeded under no circumstances. The temp., on the contrary, is responsible for the efficiency of the decompn. reaction. The higher the temp., the higher is the oxidn. potential of the digestion reagent. Recording the time temp. curve of the decompn. process the extent of sample degrdn. can be established afterwards. New developments enable recording of pressure and temp. of all **vessels** in a **microwave** oven during **digestion**. **Microwave** assisted flow **digestion** systems open up new possibilities in fully automated **sample prepn.** for element anal. For fast and almost complete **sample digestion** the temp. must be as high as possible. For this reason elevated pressure has to be applied within the flow digestion systems. For an extensive oxidn. of org. sample constituents with nitric acid temps. of $>220^{\circ}$ are necessary. **Teflon** tubes used, however, do not withstand the vapor pressure of the digestion mixt. at 220° or more. Thus new alternatives has to be found to overcome this limitation.

L12 ANSWER 42 OF 148 CA COPYRIGHT 2003 ACS on STN
 AN 128:11532 CA
 TI Development of high-pressure closed-**vessel** systems for **microwave**-assisted **sample digestion**
 AU Matusiewicz, Henryk
 CS Department of Analytical Chemistry, Politechnika Poznanska, Poznan, 60-965, Pol.
 SO Microwave-Enhanced Chemistry (1997), 353-369. Editor(s): Kingston, H. M.; Haswell, Stephen J. Publisher: American Chemical Society, Washington, D. C.
 AB A review with 61 refs. The historical development of high-pressure closed-**vessel** systems in **microwave**-assisted **sample digestion** is presented. The current state of the art, including advantages and limitations of this approach, is discussed. The construction and methodol. controls for a series of special **microwave-digestion** bombs and a universal complex **microwave-digestion** system for chem. **prepn.** for transferring the **sample** into a liq. state are described. Emphasis is laid on the completeness of the digestion, for which the use of high-pressure high-temp. focused-microwave-heated **Teflon** bomb is prerequisite. The novel prototype system uses focused microwaves, operated at 2.45 GHz, to improve digestion capability. Methodol. was developed using powd. biol. ref. material. With this new decompn. device, org. material is totally oxidized with nitric acid in a single-step procedure.

L12 ANSWER 45 OF 148 CA COPYRIGHT 2003 ACS on STN
 AN 127:85671 CA
 TI Optimized microwave preparation procedure for the elemental analysis of aquatic sediment
 AU Wen, Xiang Hua; Wu, Ling Zheng; Zhang, Yun; Chu, Yang
 CS Reasearch Center Eco-Environmental Sciences, Chinese Academy Sciences, Beijing, 100085, Peop. Rep. China

SO Fresenius' Journal of Analytical Chemistry (1997), 357(8), 1111-1115
AB This paper summarizes several key points in applying the microwave prepn. technique to the elemental anal. of aquatic sediments and reports systematic expts. in searching for an optimum microwave **prepn.** procedure for element anal. in sediment **samples**. The detn. of the elements Cu, Pb, and Cd in a std. ref. aquatic sediment sample (CRM 280, COMEUR) was achieved by 1st **digesting** the **samples** in a **microwave** oven equipped with **PFA** advanced composite **vessels**, followed by AAS measurement. The influence of **microwave** power, **digestion** time, various dissoln. reagents, and the HF removing conditions were studied. For a 0.1 g sediment **sample** the optimum microwave **prepn.** conditions were: 4-5 mL HNO₃/HF/H₂O₂ as solvent, **digesting** time 30 min with 100% **microwave** power, and evapg. the residual acid within 8 min in an open vessel at 80°. The element recovery rates with AAS measurement were <92.4-100.6%.

L12 ANSWER 47 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 127:44048 CA

TI Determination of mercury in biological and environmental samples by inductively coupled plasma mass spectrometry with the isotope dilution technique

AU Yoshinaga, Jun; Morita, Masatoshi

CS National Institute Environmental Studies, Tsukuba, 305, Japan

SO Journal of Analytical Atomic Spectrometry (1997), 12(4), 417-420

AB The accurate and precise detn. of total mercury (Hg) in biol. and environmental samples by isotope diln.-inductively coupled plasma mass spectrometry (ID-ICP-MS) is described. The precision of Hg isotope ratio (e.g., 200Hg/202Hg) measurements at the 20 ppb level was <0.5%. The deviation of the measured isotope ratios in a std. Hg soln. was <0.5% from the natural ratios. Neither spectral interferences nor matrix effects affect the accuracy and precision of the proposed Hg isotope ratio anal. Total Hg concns. in human hair and sediment CRMs were detd. by ID-ICP-MS after 202Hg addn. and acid decompn. of the samples. Solvent extn. and back-extn. was used for sediment anal. The various decompn. methods used for hair **samples**, i.e., **microwave digestion** and the **Teflon vessel double digestion** method, did not give any difference in the anal. value. The ID-ICP-MS results were better than those obtained with std. addns. or internal standardization in terms of accuracy and precision. Anal. results for human hair and sediment CRMs were in good agreement with the certified/ref. values.

L12 ANSWER 48 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 127:8843 CA

TI **Membrane solid-phase extraction** (SPE) with closed-vessel **microwave** elution for the determination of phenolic compounds in aqueous matrixes

AU Chee, Kok Kay; Wong, Ming Keong; Lee, Hian Kee

CS Department Chemistry, National University Singapore, Singapore, 119260, Singapore

SO Mikrochimica Acta (1997), 126(1-2), 97-104

AB Preconcn. of phenolic compds. was carried out with Empore C18 **membrane** disks, which were eluted with a closed-vessel **microwave** extn. (CVME) system. The optimum microwave-elution conditions were obtained by optimizing the elution solvent, elution temp., duration of elution, and vol. of elution solvent. The recoveries of 11 phenolic compds. spiked at 10 and 500 µg/L levels into water, using the optimum conditions, were all >85% with RSD 4.0-10.0%, except for phenol and 4-nitrophenol. **Extn.** of an industrial wastewater **sample** suspected of contg. phenolic compds. by SPE-CVME showed similar recoveries of phenol (the only phenolic compd. detected) when compared with LLE and C18 SPE cartridge techniques. Anal. of phenolic

compds. was carried out using liq. chromatog. coupled to a UV detection system.

L12 ANSWER 49 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 126:350942 CA

TI Evaluation of rapid **solid sample extraction** using the **microwave-assisted** process (MAP) under closed-**vessel** conditions

AU Li, Ken; Belanger, Jacqueline M. R.; Llompart, Maria P.; Turpin, Rodney D.; Singhvi, Raj; Pare, J. R. Jocelyn

CS Microwave-Assisted Processes Division, Environment Canada, Environmental Technology Centre, Ottawa, ON, K1A 0H3, Can.

SO Spectroscopy (Amsterdam) (1997), Volume Date 1996-1997, 13(1), 1-13

AB The applicability of MAP extn. and subsequent GC/MS detn. of mixts. of representative toxic substances including PAH, PCB, base-neutrals, chlorinated pesticides and substituted phenols was evaluated. Typical environmental matrixes including sand, **soil** and air **filters** were spiked, **extd.** and analyzed. Recoveries from these matrixes were acceptable (>80 %); precision was generally in the 10% (relative std. deviation) range. The method was then validated using std. ref. materials of marine sediments, harbor sediments and a creosote contaminated soil certified with PAH and PCB. Split samples were also obtained from a contract lab. and the consensus results were used to evaluate recoveries of PAH and organochlorine pesticides. The potential problem of degrdn. of thermally labile pesticides was addressed by measuring degrdn. products of DDT and Endrin after MAP extn. Spiked samples before and after the high temp./high pressure extn. process did not result in addnl. decompn. products.

L12 ANSWER 50 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 126:342911 CA

TI Total elemental analysis **digestion** method evaluation on **soils** and clays

AU Wilson, M. A.; Burt, R.; Lynn, W. C.; Klameth, L. C.

CS Soil Survey Lab., USDA Natural Resources Conservation Service, Lincoln, NE, 68508-3866, USA

SO Communications in Soil Science and Plant Analysis (1997), 28(6-8), 407-426

AB The std. **digestion** method for total elemental anal. of **soil** material by the Soil Survey Lab. (USDA Natural Resources Conservation Service) uses a 2-mL clay suspension with hydrofluoric acid (HF) in a closed **Teflon** digestion vessel (method HF-SUS). The accuracy and efficiency of elemental recovery by method HF-SUS was compared to: (a) modification of HF-SUS by use of a dried sample (method HF-DRI); (b) modification of HF-SUS by use of dried sample and HF + aqua regia (method HF + AR); (c) **sample digestion** by Li metaborate fusion (method FUS); and (d) **microwave digestion** of **samples** with HF + aqua regia in **Teflon** bombs (method MICRO). Three replications of three std. ref. materials (SRMs), fine-earth (<2 mm) from 12 soils, and the clay (<2 μ m) from 10 of those soils were analyzed. Method HF + AR shows the most consistent statistical agreement with the certified SRM values. Anal. of variance (ANOVA) indicates significant effects ($\alpha=0.05$) for method of digestion, nonsignificant effects for method times SRM and method times clay, but significant effects for method times fine-earth. Compn. and/or variability of material are significant factors in the method of digestion. Method HF + AR yields significantly higher exptl. means of Al₂O₃, Fe₂O₃, and K₂O contents and oxide recovery (summation of exptl. means for oxides of all reported elements) than all other methods.

L12 ANSWER 51 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 126:287237 CA

TI A generalized method for the determination of nickel in different **samples** by ETAAS after rapid **microwave-assisted digestion**

AU Chakraborty, Ruma; Das, Arabinda K.; Cervera, M. Luisa; De La Guardia, Miguel
CS Department of Chemistry, University of Burdwan, Burdwan, 713 104, India
SO Analytical Letters (1997), 30(2), 283-303
AB Detn. of nickel was carried out by electrothermal atomization at. absorption spectrometry (ETAAS) using unpyrocoated tubes. A generalized method was developed for quant. detn. of total nickel in different types of samples: geol., like rock, soil and sediment, environmental, like sewage sludge, and biol., like mussel tissue and rice flour, after their rapid **microwave-assisted digestion**. The systematic study of the effect of six different matrix modifiers demonstrated that, using an ashing temp. of 1000° and carrying out the atomization at 2700°, neither modifier nor background correction are necessities. The recommended procedure is based on the pressurized acid **digestion** of **samples** with aqua regia, HF and H2O2, in different steps, inside hermetically closed **teflon** reactors, heated by radiation in a microwave oven, at power levels between 330 and 550 W. After the complete dissoln. of samples, Ni is detd. by ETAAS carrying out the absorbance measurements in the peak height mode, and using aq. solns. of Ni as stds.

L12 ANSWER 54 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 126:211169 CA

TI **Microwave pressure digestion** - development, performance, and safety of a novel system of **sample preparation**

AU Kettisch, P.; Schalk, A.; Knapp, G.; Zischka, M.

CS Anton Paar KG, Graz, A-8054, Austria

SO CANAS '95, Colloquium Analytische Atomspektroskopie, Konstanz, Germany, Apr. 2-7, 1995 (1996), Meeting Date 1995, 355-359. Editor(s): Welz, Bernhard. Publisher: Bodenseewerk Perkin-Elmer, Ueberlingen, Germany.

LA German

AB An overview is given of the Multiwave system for **microwave pressure digestion** (≤80 bars for quartz reaction vessels, ≤40 bars for PFA vessels; ≤300°). The efficiency of the system is enhanced by sep. control of pressure for different reaction vessels. Reaction temp. is monitored by using an IR system. The system is appropriate for the detn. of heavy metals in food samples.

L12 ANSWER 56 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 126:152057 CA

TI Direct processing and analysis of solid and other complex samples with automatic flow injection systems

AU Zhi, Zheng-liang; Rios, Angel; Valcarcel, Miguel

CS Dep. Analytical Chem., Univ. Cordoba, Cordoba, 14004, Spain

SO Critical Reviews in Analytical Chemistry (1996), 26(4), 239-260

AB A review, with 90 refs., is given. The use of a suitable sampling or sample-processing unit connected to a flow injection system can significantly expand the scope of application of the flow injection technique by enabling the direct introduction and treatment of solid and other complex samples in a single automated system. In recent years various sample pretreatment techniques, including electrolytic dissoln., online leaching, continuous **microwave digestion**, online sterilizable **membrane dialysis/filtration**, pervaporation, dynamic gas-extn., and flow-reversal liq.-liq. extn., among others, proved useful for solving a variety of anal. problems in conjunction with flow injection systems. The principles behind these techniques and their applications in fast assay and online process monitoring are reviewed and discussed. Selected major advances in this research field are outlined.

L12 ANSWER 57 OF 148 CA COPYRIGHT 2003 ACS on STN
 AN 126:98532 CA
 TI Determination of Environmental Lead Using Continuous-Flow **Microwave Digestion** Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
 AU Beary, Ellyn S.; Paulsen, Paul J.; Jassie, Lois B.; Fassett, J. D.
 CS Chemical Science and Technology Laboratory, National Institute of Standards and Technology, Gaithersburg, MD, 20899, USA
 SO Analytical Chemistry (1997), 69(4), 758-766
 AB A com. flow-through microwave system was successfully applied as an alternative **sample prepn.** procedure for lead detns. using isotope diln. inductively coupled plasma quadrupole mass spectrometry. Sample is introduced as a slurry and then injected into a carrier stream which is continually flowing through the system. As configured, the sample dissoln. is sequential, taking ~10 min/sample. This closed system is easy to use and produces low blanks, thus providing a viable alternative to the direct introduction of untreated samples, which can degrade anal. results. Leaves, air **filters**, urine, sludge, dust, and paint std. ref. materials previously certified for lead using isotope diln. mass spectrometry (IDMS) were used to evaluate the accuracy of this automated **sample prepn.** device. Lead concns. in the nanograms to micrograms per g range were accurately detd. when compared with the certified value and previous IDMS results.

L12 ANSWER 58 OF 148 CA COPYRIGHT 2003 ACS on STN
 AN 126:79426 CA
 TI Wastewater treatment system
 IN Marchesseault, Guy; Rummler, John; Sauk-Schubert, Heinz; Beal, Thomas; Mchenry, Steven
 PA Wastech International, Incorporated, USA; Marchesseault, Guy; Rummler, John; Sauk-Schubert, Heinz; Beal, Thomas; Mchenry, Steven
 SO PCT Int. Appl., 110 pp.
 PI WO 9637440 A1 19961128 WO 1996-US6964 19960522
 US 5725762 A 19980310 US 1995-445726 19950522
 US 5843304 A 19981201 US 1995-445686 19950522
 PRAI US 1995-445686 A 19950522
 AB A treatment system is provided which can treat solid materials and/or liq. materials. Preferably, a separator initially separates liq. and solid materials, and the **solid** materials are deposited in a **microwave reaction** chamber to be subjected to **microwaves**. The liqs. exiting the separator are preferably also passed through a **filter** assembly, which retains any suspended particles remaining in the liq., and subjects the particles to microwaves. Exhaust **filter** can also be provided for the **microwave reaction** chamber.

L12 ANSWER 62 OF 148 CA COPYRIGHT 2003 ACS on STN
 AN 125:157190 CA
 TI Use of a **microwave** oven for **digestion** of analytical **samples** before determination of metals by atomic absorption spectrometry (AAS)
 AU Przygoda, Dorota; Dittwald, Ewa; Samsonowska, Katarzyna; Kalinowski, Krzysztof
 CS Instytut Chemii Przemyslowej, Warsaw, Pol.
 SO Chemik (1996), 49(5), 125-126
 LA Polish
 AB Procedures for **microwave digestion** of selected vegetable materials, cosmetics, salts and esters of higher fatty acids, galvanic wastes and catalysts are elaborated. For the **digestion** and mineralization of **samples** a CEM company microwave oven type MDS-2000 equipped with translucent **PFA** containers with TPFE inserts was used. In the applications mentioned above a considerable time redn. were obtained, esp. in the detn. of metals in

mixts. with low- and high-boiling org. compds. contg. Cr salts of org. acids.

L12 ANSWER 67 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 124:337083 CA

TI **Microwave digestion** using dual **PTFE containers** for analysis of trace elements in small amounts of biological samples

AU Mizushima, Ryuji; Yonezawa, Mitsuru; Ejima, Akiko; Koyama, Hiroshi; Satoh, Hiroshi

CS Nippon General Trading Co., Tokyo, 103, Japan

SO Tohoku Journal of Experimental Medicine (1996), 178(1), 75-9

AB The anal. of trace elements in biol. samples is essential to extend our knowledge on human health and disease. Inductively coupled plasma mass spectrometry (ICP-MS) makes it possible to simultaneously det. these elements in trace amts. Before anal., however, biol. samples such as organs and tissues must be liquefied and extra org. materials must be decompd. by acid digestion. We established a method of **microwave digestion** using dual **PTFE containers** to minimize the amt. of samples. Samples (35-45 mg) of std. ref. materials, bovine liver (1577a, NIST) and fish flesh (MA-A-2, IAEA), were weighed in **PTFE-PFA** vials and a small amt. of nitric acid (0.5 mL) was added. The vials were sealed and two **PTFE-PFA** vials were placed in a **PTFE-TFM** vessel contg. 6 mL of pure water. Then the vessels were placed in a rotor and the **samples** were **digested** for 38 min in a **microwave** oven according to a pre-set program. After the program was completed, the samples were analyzed by ICP-MS. The detd. values of elements of the **microwave-digested samples** matched the certified values of the std. ref. materials. Therefore, the digestion using dual containers was successfully applied to small samples.

L12 ANSWER 68 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 124:330955 CA

TI Application of inductively coupled plasma mass spectrometry for total metal determination in silicon-containing solid samples using the microwave-assisted nitric acid-hydrofluoric acid-hydrogen peroxide-boric acid digestion system

AU Wu, Shaole; Zhao, Yu-Hui; Feng, Xinbang; Wittmeier, Adolph

CS Environ. Chem., Alberta Environ. Cent., AB, T9C 1T4, Can.

SO Journal of Analytical Atomic Spectrometry (1996), 11(4), 287-96

AB The **microwave**-assisted conventional HNO₃-HF-H₂O₂-H₃BO₃ **digestion** system was explored for direct ICP-MS anal. of total metals in silicon-contg. solid samples. In closed **Teflon PFA vessels** under **microwave** heating with temp./pressure regulation, a 0.25 g portion of **sample** contg. ≤33% silicon was **digested** in 1.5 mL of HF (48%), 5 mL of concd. HNO₃ and 2 mL of H₂O₂ (30%), followed by a 2nd digestion stage with 12 mL of 5% m/v boric acid. The amt. of HF used was approx. twice the stoichiometric requirement for 33% silicon contained in the samples, assuming all silicon exists as silica. The amt. of boric acid used was the stoichiometric requirement for the HF added. With a sample diln. factor of 2000 (v/m), the end soln. contained ≤0.17% total dissolved solids for ICP-MS anal. The ICP-MS system was calibrated by the method of external stds. prepd. in reagent blank solns. with In as the internal std. The sensitivity of the In signal in a 5-8 h operation was only decreased by 5-10%. Background interferences from the developed is simple and rugged and suitable for routine anal. of at least 25 elements: Al, Ag, As, Ba, Bi, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Ni, Pb, Sb, Sn, Sr, Th, Ti, Tl, U, V and Zn. The recoveries for most of these elements in std. ref. materials including coal fly ash, urban particulate matter, sediment and soil were within 90-110%, and the relative sediment and soil were within 90-110%, and the relative std. deviations were within 5%.

The method cannot det. B and is not suitable coal fly ash, urban particulate matter, sediment and soil were within 90-110%, and the relative std. deviations were within 5%. The method cannot det. B and is not suitable for Hg detn. The detection limits for Be and Se detn. are not adequate.

L12 ANSWER 69 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 124:330921 CA

TI The comparison of **sample preparation** techniques for the determination of technetium-99 in pure uranium compounds and subsequent analysis by inductively coupled plasma-mass spectrometry (ICP-MS)

AU Makinson, Peter R.

CS Chemical and Metallurgical Services Department, British Nuclear Fuels plc., Preston/Lancashire, PR4 0XJ, UK

SO ASTM Special Technical Publication (1995), STP 1291 (Applications of Inductively Coupled Plasma-Mass Spectrometry to Radionuclide Determinations), 7-19

AB In this presentation, a procedure for the detn. of ng/g levels of technetium-99 in uranium dioxide powder, hydrolyzed uranium hexafluoride (Hex), and uranyl nitrate liquor (UNL) is discussed, and three methods for the dissoln. of uranium dioxide are compared. Digestion of uranium dioxide using concd. nitric acid in a stoppered glass tube gives variable results. A concd. hydrochloric acid dissoln. using **microwave digestion** in sealed TFE-fluorocarbon **PFA digestion** vessels gives reproducible results, as does the digestion using dil. nitric acid in open beakers. The effects of the isobaric interference of ruthenium-99 and the mol. interference of 98MoH and the use of suitable algorithms to correct for such interferences are discussed. Compensation of signal suppression due to the uranium matrix is made using matrix-matched calibration std. solns. and instrumental drift is monitored and cor. by the addn. of rhodium as an internal std. A detection limit (2 s) of 0.3 ng g⁻¹ U is achieved using a soln. contg. 2 g of uranium per L and a 30-s per isotope acquisition time, using peak jump mode.

L12 ANSWER 73 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 124:210840 CA

TI Trace metal analysis of coal fly ash collected plain and on a quartz fiber **filter**

AU Laitinen, T.; Revitzer, H.; Tolvanen, M.

CS VTT, Chem. Technol., Environ. Technol., Espoo, FIN-02044, Finland

SO Fresenius' Journal of Analytical Chemistry (1996), 354(4), 436-41

AB A simple, uniform procedure has been developed for **microwave-based digestion** of fly ash **samples** collected from the hoppers of an electrostatic precipitator (ESP), or collected from flue gases on a heat-resistant quartz fiber **filter** and for subsequent at. absorption spectrometric anal. of trace metals (Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, V and Zn). HNO₃, HCl, HF and mixts. have been tested as digestion acids. The combination of HNO₃ and HF has been found to be efficient for the **digestion** of fly ash **samples** with and without quartz fiber **filter** material. In spite of the complicated matrix, results with satisfactory accuracy and precision (relative std. deviation below 10% for most of the elements) have been obtained. In the case of anal. of fly ash together with a quartz fiber **filter**, the calibration curve has to be detd. by std. addn. to a blank soln. contg. a dissolved blank quartz fiber **filter**.

L12 ANSWER 74 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 124:183999 CA

TI Multi-element analysis of airborne particulate matter by various spectrometric methods after **microwave digestion**

AU Wang, Chu-Fang; Yang, Jenq-Yann; Ke, Cheng-Hsiung

CS Institute of Nuclear Science, National Tsing Hua University, Hsinchu, 30043, Taiwan

SO Analytica Chimica Acta (1996), 320(2-3), 207-16

AB An anal. method using **microwave digestion** procedure in combination with various spectrometric methods, including at. absorption spectrometry, inductively coupled plasma at. emission spectrometry, and inductively coupled plasma mass spectrometry, was developed for the multi-element detn. in airborne particulate matter collected on **PTFE filters** by a dichotomous sampler. In order to achieve more sensitive and rapid multi-element analyses, special **PTFE-lined** digestion vessels were used. It was found that complete digestion of airborne particulates with an acid mixt. of HNO₃-HClO₄-HF (3:7:1, vol./vol./v) can be achieved in the **microwave-irradiated** closed **vessel** system and direct spectroscopic measurement of the **digested sample** after appropriate diln. A recovery study was conducted using a multi-element std. and NIST Std. Ref. Material 1648 Urban Particulate. Sixteen major, minor, and trace elements in airborne particulate matter were detd.

L12 ANSWER 76 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 123:295015 CA

TI Analytical procedures on multi-element determinations of airborne particles for receptor model use

AU Wang, C. F.; Chang, E. E.; Chiang, P. C.; Aras, N. K.

CS Institute Nuclear Science, National Tsing Hua University, Hsinchu, Taiwan

SO Analyst (Cambridge, United Kingdom) (1995), 120(10), 2521-7

AB A multistage anal. procedure is developed in this investigation to det. 22 elements (Al, As, Ba, Ca, Cd, Cl, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, S, Sb, Si, Sr, Ti, V, and Zn) on loaded **PTFE-filter** samples of atm.-particulate matter collected by dichotomous samplers. It includes a direct x-ray fluorescence measurement followed by instrumental neutron activation anal. (INAA) or **microwave acid digestion** of the **sample** for spectroscopic anal. ICP-MS was employed for elemental anal. after the digestion. A combination of different anal. methods and std. operational procedures were developed to meet the requirements for receptor model anal. The quality assurance program includes the intercomparison with the use of alternative methods for accuracy and precision control.

L12 ANSWER 79 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 123:217229 CA

TI **Microwave digestion** and alkali fusion procedures for the determination of the platinum-group elements and gold in geological materials by ICP-MS

AU Totland, Marina M.; Jarvis, Ian; Jarvis, Kym E.

CS School of Geological Sciences, Kingston University, Penrhyn Road, Kingston-upon-Thames, Surrey, KT1 2EE, UK

SO Chemical Geology (1995), 124(1-2), 21-36

AB Inductively coupled plasma-mass spectrometry (ICP-MS) is an ideal technique for detg. the Pt-group elements (PGE's) and Au, with rapid data acquisition and low detection limits of 0.03-0.22 ng mL⁻¹. Two methods are described for the **prepn.** of geol. **samples** using **microwave digestion** prior to detn. of the PGE's and Au by ICP-MS. In one method, 0.5-g samples are dissolved in sealed all-**PFA microwave digestion vessels** using HNO₃-HCl-HF-HClO₄ acids. Samples are transferred to open **PTFE** beakers and evapd. to incipient dryness, final solns. being taken up in 1 M HCl prior to anal. The method was evaluated using a suite of well-characterized international ref. materials (RM's). In some cases, the method resulted in a complete digestion and quant. data were obtained for Rh, Pd, Ir, Pt and Au, although insufficiently low limits of detn. precluded the detn. of all five elements in all materials. In other cases, an insol. residue remained and recoveries

of the PGE's and Au were dependent on the element concerned and the mineralogy of each sample. A 2nd method employs 1-g **samples** and **microwave digestion** with aqua regia-HF, in higher-pressure **Utem-jacketed Teflon PFA** sealed-vessels. **Samples** are subsequently evapd. to near dryness, **digested** in 0.5M HCl, filtered, and the insol. residues are fused with small quantities of 1:1 Na₂O₂+Na₂CO₃ or Na₂O₂, before being dissolved in 0.5M HCl. The combined solns. are analyzed by ICP-MS. Data obtained for a wide range of RM's showed good agreement with ref. values. Both methods provide viable means of quantifying Ru, Rh, Pd, Ir, Pt and Au in mineralized samples, but both are limited by modest lower limits of detn. in samples of 0.2-1 µg g⁻¹. Only the combined **microwave digestion**-minifusion technique yields fully quant. data for samples contg. refractory minerals.

L12 ANSWER 80 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 123:142049 CA

TI Manganese and Zinc Analysis in Milk by **Microwave Oven Digestion** and Platform Graphite Furnace Atomic Absorption Spectrometry

AU de la Fuente, Miguel Angel; Guerrero, Gonzalo; Juarez, Manuela

CS Instituto del Frio (CSIC), Ciudad Universitaria s/n, Madrid, 28040, Spain

SO Journal of Agricultural and Food Chemistry (1995), 43(9), 2406-10

AB A conventional **microwave oven acid digestion** system employing **PTFE** reactors was tested for **sample digestion** prior to the anal. of Mn and Zn in milk. The **digestion** procedure was based on **sample** dissoln. with nitric acid using **microwave** heating in closed **vessels**. Sample treatment time in the microwave oven was reduced to 15 min. The resulting solns., analyzed for Zn by flame at. absorption spectrometry (FAAS) and by platform furnace at. absorption spectrometry with Zeeman background correction for Zn and Mn, showed good agreement with the certified milk values (SRM-1549 nonfat milk powder, National Institute of Stds. and Technol.), and results were comparable to those obtained by dry mineralization in real samples. The precision of the method was about 6% for Mn and 5% for Zn by graphite furnace at. absorption spectrometry (GFAAS) with limits of detection of 0.07 and 0.28 µg/L, resp. The digestion procedure can be used to det. Zn by FAAS and Mn by GFAAS in milk anal. Using GFAAS, Zn can also be measured in milk fractions where it is found in smaller concns.

L12 ANSWER 83 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 123:39703 CA

TI Microwave decomposition for airborne particulate matter for the determination of trace elements by inductively coupled plasma mass spectrometry

AU Wang, C. F.; Chen, W. H.; Yang, M. H.; Chiang, P. C.

CS Inst. Nucl. Sci., Natl. Tsing Hua Univ., Hsinchu, 30043, Taiwan

SO Analyst (Cambridge, United Kingdom) (1995), 120(6), 1681-6

AB A **microwave digestion** method utilizing an acid mixt. of HNO₃-HClO₄-HF (3 + 5 + 2 vol./vol.) in closed vessels was developed for the detn. of major, minor and trace elements in very small amts. of airborne particulate matter collected on **Teflon filters** by a dichotomous sampler, and was tested on NIST Std. Ref. Material (SRM 1648 Urban Particulate Matter). Analyses were carried out by inductively coupled plasma mass spectrometry (ICP-MS). Sixteen elements (Al, As, Ba, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V and Zn) were detd. The results generally showed high reproducibility and good agreement with the certified values, except for As and Cr. The feasibility of applying the **microwave digestion** pre-treatment with ICP-MS to real samples was also examd. It was demonstrated that the suggested method is more suitable than conventional methods for real sample anal.

L12 ANSWER 85 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 121:270645 CA
TI Application of pressurized **sample preparation** methods for analysis of steels and copper alloys
AU Borszeki, Janos; Halmos, Pal; Gegus, Erno; Karpati, Peter
CS Dep. Anal. Chem., Univ. Veszprem, Veszprem, H-8201, Hung.
SO Talanta (1994), 41(7), 1089-93
AB Pressurized **sample prepn.** devices (High Pressure Asher, Pressurized **Microwave Digestion** system, compared with a PTFE decompn. vessel) were used to dissolve certified metal alloy samples (steel, Cu) for ICP anal. Based on the results of the anal. both up-to-date devices can be advantageously applied to quickly and quant. dissolve metal alloy samples. To dissolve the samples, two different kinds of acid mixts. (A: nitric and HCl; B: nitric and hydrochloric and sulfuric and H₃PO₄) were used. The **sample prepn.** is simpler and less time-consuming than the earlier commonly used methods, sample loss and degree of contamination are also reduced. Steel samples contg. W, Ti and Nb (<0.5%) can only be analyzed using a mixt. of the four acids. By dissolving steel samples in the nitric and HCl mixt., the concn. of their most common elements (Cr, Ni, Mn, V, Cu) as well as their S and P content can be detd. Cu alloy samples can be dissolved quickly by the pressurized microwave decompn. device using HCl and dild. (1:1) HNO₃.

L12 ANSWER 93 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 120:123820 CA
TI Development of a high pressure/temperature focused microwave heated **teflon** bomb for **sample preparation**
AU Matusiewicz, Henryk
CS Dep. Anal. Chem., Politech. Poznanska, Poznan, 60-965, Pol.
SO Analytical Chemistry (1994), 66(5), 751-5
AB A new high pressure/temp. TFM-**Teflon** bomb for anal. **sample prepn.** is described. The novel prototype system uses focused microwaves, operated at 2.45 GHz, to improve digestion capability. Up to 100-W microwave power can be concd. into a single polymer deriv. vessel contg. sample and HNO₃. Methodol. was developed using powd. biol. ref. material. The feasibility of using H₂O for in situ vessel cooling was studied. The residual C content of bovine liver sample was detd. by coulometry after combustion in an O stream to evaluate the effectiveness of the decompn. procedure. With this new decompn. device, org. material is totally oxidized with HNO₃ in a single-step procedure. The **sample prepn.** time is ~10 min (including subsequent cooling time and prepn. of the final soln.).

L12 ANSWER 95 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 119:208879 CA
TI High-pressure microwave dissolution of ceramics prior to trace metal determinations by microwave induced plasma atomic emission spectrometry
AU Matusiewicz, Henryk
CS Dep. Anal. Chem., Politech. Poznanska, Poznan, 60-965, Pol.
SO Mikrochimica Acta (1993), 111(1-3), 71-82
AB A com. lab. **microwave acid digestion** system was evaluated for the acid dissoln. of ceramic powders (Al₂O₃, AlN, BN and Si₃N₄) prior to the detn. of their trace element content by microwave induced plasma at. emission spectrometry. Newly designed vessels, capable of withstanding internal pressures of over 110 bar, provide rapid and satisfactory results for sample dissoln. **Sample prepn.** time was approx. 30 min (including the subsequent cooling time and prepn. of the final soln.). Results from conventional stainless-steel acid digestion vessel (**Teflon** bomb) dissoln. are compared with the microwave bomb results of microwave plasma at. emission spectrometry.

L12 ANSWER 100 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 118:3368 CA
TI Factorial design approach to microwave dissolution
AU Mohd, A. A.; Dean, J. R.; Tomlinson, W. R.
CS Sch. Appl. Sci., Inst. Teknol. Mara, Shah Alam, 40000, Malay.
SO Analyst (Cambridge, United Kingdom) (1992), 117(11), 1743-8
AB A fractional factorial design was used to explore the variables that affect **microwave** dissoln. using perfluoroalkoxy (PFA)-**Teflon digestion vessels**. Optimum operating conditions for Nat. Research Council of Canada, certified ref. material TORT-1 Lobster Hepatopancreas and Nat. Inst. of Stds. and Technol., Std. Ref. Material (SRM) 1575 Pine Needles, were obtained by this procedure. The optimum conditions found for each variable are: 0.25 g sample mass, 6 mL concd. HCl, 6 mL concd. HNO₃, 3 mL concd. HF, and 90% microwave power for a total dissoln. time of 15 min. Results of Ca, Fe, Cu, and Zn in TORT-1 and Ca and Fe in SRM 1575 were in agreement with the certified values.

L12 ANSWER 104 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 117:107632 CA
TI **Microwave digestion** of biological **samples** with acid mixture in a closed double **PTFE** vessel for metal determination by "one-drop" flame atomic absorption spectrometry
AU Kojima, I.; Kato, A.; Iida, C.
CS Lab. Anal. Chem., Nagoya Inst. Technol., Nagoya, 466, Japan
SO Analytica Chimica Acta (1992), 264(1), 101-6
AB A microwave-heat system using a closed double **PTFE** vessel with a polypropylene jacket was evaluated for rapid **sample prepn.** for the detn. of trace elements in biol. samples. The **microwave-assisted digestion** was based on **sample** dissoln. with a HNO₃-HClO₄-HCl-HF mixt. The simultaneous **digestion** of six **samples** including one blank was completed within 15 min even with very mild heating. The presence of an alk. soln. between the **PTFE** vessels was very effective in aiding the digestion and absorption of evolved acid gases. Six metals (Ca, Cu, Fe, Mg, Mn and Zn) in std. ref. materials, NIST-SRM 1577 Bovine Liver, NIES-CRM No. 1 Pepperbush, No. 6 Mussel, No. 5 Human Hair, No. 7 Tea Leaves and No. 9 Sargasso, were analyzed by one-drop flame at. absorption spectrometry. Good agreement of the anal. results with the certified values was obtained.

L12 ANSWER 110 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 115:202693 CA
TI Rapid stopped-flow **microwave digestion** system
AU Karanassios, Vassili; Liu, F. H.; Liu, B.; Salin, Eric D.
CS Dep. Chem., McGill Univ., Montreal, QC, H3A 2K6, Can.
SO Journal of Analytical Atomic Spectrometry (1991), 6(6), 457-63
AB A prototype system for stopped-flow **microwave-assisted wet digestions** has been developed. A coiled **Teflon** perfluoroalkoxy tube serves both as a **sample** container and as a **digestion** vessel. A sample plug, consisting of a water slurry mixed with an acid mixt., is pumped into the coil. Sample flow is stopped, the coiled tube is sealed (by closing an input and an output valve), and microwave power is applied for 2 min for **digestion** of the **sample**. Methodol. was developed using powd. botanical ref. samples and was tested with powd. botanical and biol. ref. materials. The digests were analyzed by inductively coupled plasma at. emission spectrometry. In addn. to comparisons with certified values, the results were compared with those obtained by conventional open-vessel hot-plate **digestions**, by open-vessel **microwave digestions**, and by **digestions** taking 32 min by using the coiled tube system. Precise, and in many instances, quant. digestions were obtained using a net digestion time of 2 min. Elemental recoveries were

sample type and **digestion** time dependent and were comparable with, and sometimes, superior to those obtained when using a 3-h long hot-plate digestion. In this preliminary study, characteristics, limitations, and future directions are discussed.

L12 ANSWER 115 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 115:63514 CA

TI Vapor-phase acid digestion of inorganic and organic matrixes for trace element analysis using a microwave heated bomb

AU Matusiewicz, Henryk

CS Dep. Anal. Chem., Politech. Poznanska, Ponznani, 60-965, Pol.

SO Journal of Analytical Atomic Spectrometry (1991), 6(4), 283-7

AB A vapor-phase **microwave** pressure **digestion** technique employing a special **polytetrafluoroethylene**-based microsampling device was evaluated for the acid **digestion** of marine sediment and biol. tissue **samples** prior to the detn. of their trace and minor element content. Inorg. and org. constituents are almost completely solubilized by vapor-phase attack (with an HNO₃-HF mixt. for the marine sediment and HNO₃ for the marine biol. tissue) in a perfluoroalkoxy-**Teflon** pressure bomb. The residue was taken up in 0.5 mol dm⁻³ HNO₃ and analyzed by flame and electrothermal at. absorption spectrometry. Good agreement between the results and certified values for 15 elements was found. The **sample prepn.** time was approx. 45 min for the biol. tissue and 90 min for the sediment (including the subsequent cooling time and prepn. of the final soln.).

L12 ANSWER 118 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 114:239386 CA

TI Application of the **microwave** acid **digestion** method to the decomposition of rock **samples**

AU Suzuki, Toshitaka; Sensui, Mayumi

CS Fac. Sci., Yamagata Univ., Yamagata, 990, Japan

SO Analytica Chimica Acta (1991), 245(1), 43-8

AB The **microwave** acid **digestion** method was applied to the decompn. of rock samples and optimum conditions were investigated. Samples of 10-100 mg were decompd. by changing the amt. and compn. of acid, heating time, and no. of reheating steps, and then the concns. of Si, Fe, Mn, Na, K and Mg in these samples were measured. The concns. agreed with reported values when 10 mg of sample were decompd. by heating for 60 s with 0.3 mL of concd. HNO₃ and 0.1 mL of concd. HF. Similarly, 100 mg of sample were also decompd. successfully by heating for 45-110 s with 0.3-1.0 mL of concd. HNO₃ and 0.4-0.7 mL of concd. HF. It is concluded that the **microwave** acid **digestion** method decomp. rock **samples** with a very short heating time and with small amt. of reagents compared with methods using conventional sealed **PTFE** vessels, which require several hours for the heating step and several milliliters of reagents.

L12 ANSWER 125 OF 148 CA COPYRIGHT 2003 ACS on STN

AN 113:223519 CA

TI Practical options for **microwave** digestions

AU Kimber, Graham M.; Kokot, Serge

CS Queensland Univ. Technol., Brisbane, Australia

SO TrAC, Trends in Analytical Chemistry (1990), 9(6), 203-7

AB A review with 28 refs. Some options for the use of **microwave** ovens to facilitate acid **digestion** of various anal. **samples** are discussed. The use of inexpensive domestic ovens in conjunction with com. available **polytetrafluoroethylene** digestion vessels is highlighted. Various lab. practices are discussed and examples of successful acid digestion recipes are reported.

L12 ANSWER 129 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 113:108340 CA
TI **Microwave acid digestion** of geological and biological standard reference materials for trace element determination by inductively coupled plasma-mass spectrometry
AU Noeltner, Thomas; Maisenbacher, Peter; Puchelt, Harald
CS Inst. Petrogr. Geochem., Univ. Karlsruhe, Karlsruhe, D-7500/1, Germany
SO Spectroscopy (Duluth, MN, United States) (1990), 5(4), 49-53
AB Rapid acid digestions of a variety of geol. and biol. **samples** are possible using **microwave digestion** techniques. To meet the anal. requirements and the extremely low blank background values necessary for accurate trace and ultratrace element detn. with inductively coupled plasma-mass spectrometry (ICP-MS), special **polytetrafluoroethylene**-based digestion vessels were used. The particular features of the **microwave digestion** system, including the automatic capping module and the gas exhaust module, were set up to minimize the possibility of error due to sample contamination. Anal. procedures and results of the expts. are reported.

L12 ANSWER 138 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 111:93236 CA
TI Trace element analysis of biological material following pressure **digestion** with nitric acid-hydrogen peroxide and **microwave** heating
AU Matusiewicz, Henryk; Sturgeon, Ralph E.; Berman, Shier S.
CS Div. Chem., Natl. Res. Counc. Canada, Ottawa, ON, K1A 0R9, Can.
SO Journal of Analytical Atomic Spectrometry (1989), 4(4), 323-7
AB A com. **microwave acid-digestion** system employing three types of closed vessels, a pressure-relief **PFA-Teflon** bomb (A); a pressure-relief Berghof all **PTFE** bomb (B); and a completely closed Parr **microwave acid digestion** bomb (C), was evaluated for **sample digestion** prior to the detn. of trace elements in a biol. tissue. The **digestion** procedure was based on **sample** dissoln. with an HNO₃-H₂O₂ mixt. using microwave heating. The resulting solns., analyzed by flame and graphite furnace at. absorption spectrometry, showed good agreement for all three closed-vessel techniques. **Sample prepn.** time was approx. 10 min for vessels B and C, and 15 min for vessel A (including subsequent cooling time and prepn. of the final soln.).

L12 ANSWER 148 OF 148 CA COPYRIGHT 2003 ACS on STN
AN 105:130099 CA
TI Microwave energy for acid decomposition at elevated temperatures and pressures using biological and botanical samples
AU Kingston, H. M.; Jassie, L. B.
CS Cent. Anal. Chem., Natl. Bur. Stand., Gaithersburg, MD, 20899, USA
SO Analytical Chemistry (1986), 58(12), 2534-41
AB A closed **vessel microwave digestion** system is described. In situ measurement of elevated temps. and pressures in closed Telfon **PFA** vessels during acid decompn. of org. samples is demonstrated. Temp. profiles for the acid decompn. of biol. and botanical std. ref. materials are modeled by the dissolving acid. Microwave power absorption of nitric, hydrofluoric, sulfuric, and hydrochloric acids is compared. An equation is applied to acid microwave interactions to predict the time needed to reach target temps. during sample dissoln. Reaction control techniques and safety precautions are recommended.

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